Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2-(3-Ethyl-2,3,4,5-tetrahydro-4-oxo-2-thioxo-4-thiazolidin-5-ylidene)-1,2-dihydroacenaphthylen-1-one

T. V. Sundar,[a] V. Parthasarathi,[b]* S. García-Granda,[c] Abhinandan Jain[d] and R. T. Pardasani[d]

[a]Department of Physics, National College, Tiruchirappalli 620 001, India, [b]Department of Physics, Bharathidasan University, Tiruchirappalli 620 024, India, [c]Departamento de Química Física y Analítica, Universidad de Oviedo, 33006 Oviedo, Spain, and [d]Department of Chemistry, University of Rajasthan, Jaipur 302 004, India

Correspondence e-mail: vpsarati@yahoo.com

Received 4 November 2003
Accepted 11 November 2003
Online 22 November 2003

In the title compound, $C_{17}H_{11}NO_2S_2$, the thiazolidinone moiety is almost coplanar with the acenaphthylene moiety. The molecular packing is influenced mainly by weak intra- and intermolecular C—H···O interactions.

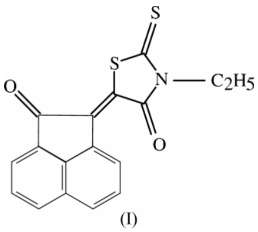
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.052
wR factor = 0.151
Data-to-parameter ratio = 11.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

We are interested in the X-ray structure determination of some novel hetero-polycyclic compounds, since they can be used as precursors for the total synthesis of naturally occurring alkaloids (Grundon, 1988). The structure determination of the title compound, (I), is the third in a series involving the acenaphthylene group.



(I)

The C—C distances in the acenaphthylene group range from 1.360 (6) (C4—C5) to 1.513 (5) Å (C1—C12) and the C—C—C angles range from 106.1 (3) (C11—C9—C12) to 123.0 (4)° (C6—C7—C8). These values compare well with the corresponding values in our previous structures, *viz*. (*rac*-*RS*,7*RS*,8*SR*)-spiro-[7-methoxycarbonyl-1-aza-3-thia-bicyclo-[3.3.0]-octane-8,1′-acenaphthylen]-2′-one (Sundar *et al.*, 2002) and (*rac*-2*SR*,3*RS*,5*SR*)-spiro-[3-methoxycarbonyl-1-aza-bicyclo[3.3.0]-octane-2,1′-acenaphthylen]-2′-one (Sundar *et al.*, 2003).

The dihedral angle between the least-squares planes of the thiazolidinone and acenaphthylene moieties is 3.23 (11)°. The geometry around the N atom is planar trigonal (Table 1).

In (I), atom C3 acts as a donor for a weak intermolecular C—H···O interaction with carbonyl atom O2 of the thiazolidinone moiety of an adjacent molecule. This interaction links the molecules into a chain which runs parallel to the *a* axis and has graph-set motif $C(8)$. Atom C7 is involved in a weak intermolecular C—H···O interaction with another carbonyl atom, O1, of an acenaphthylene moiety of an adjacent molecule. This interaction also links the molecules into a chain that runs parallel to the *a* axis and has graph-set motif $C(7)$. The above two interactions combine to form a ring motif $R_2^2(13)$ (Fig. 2 and Table 2) (Bernstein *et al.*, 1995). An intramolecular C—H···O interaction is observed between atoms C8 and O2, forming an $S(7)$ loop.

DOI: 10.1107/S160053680302600X

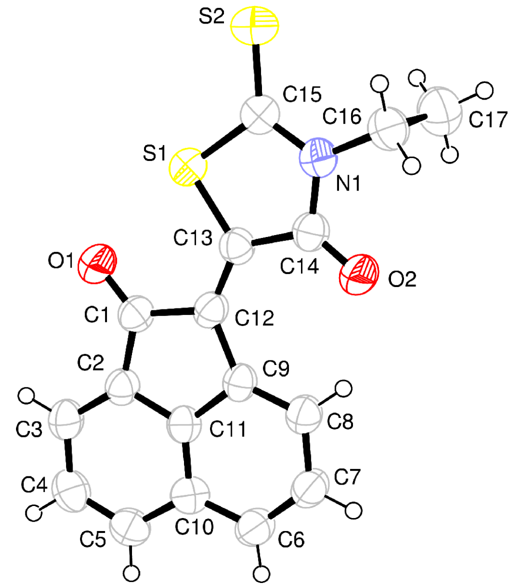


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

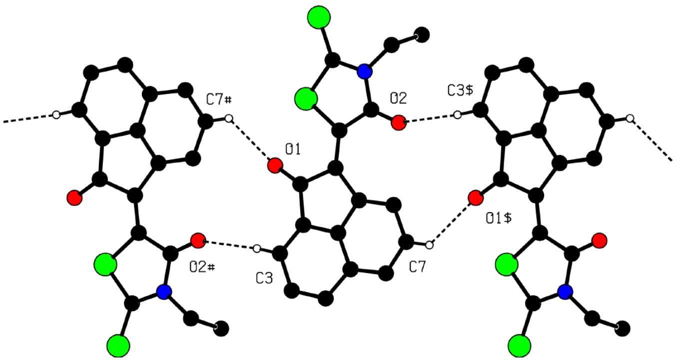


Figure 2
A view of the molecular chain in (I). The hash (#) and dollar ($) symbols correspond to symmetry codes (i) and (ii), respectively, in Table 2.

Experimental

A reaction mixture of acenaphthacene-1,2-dione (0.364 g, 2 mmol) and 3-ethylrhodanine (0.266 g, 2 mmol) was refluxed for 6 h in absolute ethanol (40 ml). The reaction was monitored by thin-layer chromatography until complete consumption of the reactants. The crude product was subjected to column chromatography over a silica gel column using solvents of increasing polarity. Compound (I) was obtained from a petroleum ether–chloroform (1:2) mixture as an orange solid (yield 0.48 g, 74%, m.p. 483 K). For crystallization, the diffusion method was adopted. A small quantity (0.24 g) of the compound was dissolved in chloroform (3 ml). *n*-Hexane (15 ml) was then layered carefully down the side of the test-tube on to the solution. The volume ratio of solvent/precipitant taken was 1:5. The test tube was then plugged with cotton wool and left to stand undisturbed for 48 h. As the precipitant (*n*-hexane) diffused into the solution, crystals were formed at the interface. The solvent was decanted off and the orange needle-shaped crystals were dried *in vacuo*.

Crystal data

$C_{17}H_{11}NO_2S_2$
$M_r = 325.39$
Monoclinic, $P2_1/c$
$a = 7.382$ (5) Å
$b = 26.375$ (5) Å
$c = 7.511$ (5) Å
$\beta = 92.911$ (5)°
$V = 1460.5$ (14) Å^3
$Z = 4$

$D_x = 1.480$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 2248 reflections
$\theta = 2.0$–68.0°
$\mu = 3.36$ mm^{-1}
$T = 293$ (2) K
Prism, orange
0.12 × 0.11 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
φ scans with κ offsets
Absorption correction: none
4416 measured reflections
2338 independent reflections
1427 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.026$
$\theta_{\rm max} = 69.2$°
$h = -8 \rightarrow 8$
$k = -31 \rightarrow 31$
$l = -9 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.151$
$S = 1.01$
2338 reflections
200 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0792P)^2 + 0.1394P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.25$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (°).

C15—N1—C14	117.4 (3)	C14—N1—C16	119.7 (3)
C15—N1—C16	122.9 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2$^{\rm i}$	0.93	2.50	3.423 (5)	172
C7—H7···O1$^{\rm ii}$	0.93	2.59	3.290 (5)	132
C8—H8···O2	0.93	2.23	2.952 (4)	134

Symmetry codes: (i) $x-1, \frac{1}{2}-y, z-\frac{1}{2}$; (ii) $1+x, \frac{1}{2}-y, \frac{1}{2}+z$.

The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å) with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C). All remaining H atoms were placed in geometrically idealized positions (C—H = 0.93–0.97 Å) and were constrained to ride on their parent non-H atoms with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). Owing to the poor quality of the crystal, some higher order sets of frames contain very weak reflections which could not be integrated. As a consequence, the data set is not complete (86%), since some higher order reflections are missing.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *DIRDIF*99 (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

TVS thanks the UGC, India, for a grant in the form of a Minor Research Project (F.MRP-039/02, Link No. 1030). SGG thanks the Spanish MCyT (BQU2000-0219) and FICYT(PR-01-GE-4) for financial support.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Beurskens, P. T., Beurskens, G., de Gelder, R., García-Granda, S., Gould, R. O., Israel, R. & Smits, J. M. M. (1999). The *DIRDIF*99 Program System. Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Grundon, M. F. (1988). *The Alkaloids*, Vol. 32, edited by A. Brossi. San Diego: Academic Press.

Nonius (1997–2000). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Sundar, T. V., Parthasarathi, V., Alvarez-Rúa, C., García-Granda, S., Saxena, A., Pardasani, P. & Pardasani, R. T. (2002). *Acta Cryst.* E**58**, o1405–o1407.

Sundar, T. V., Parthasarathi, V., Alvarez-Rúa, C., García-Granda, S., Sharma, I. & Pardasani, R. T. (2003). *Acta Cryst.* E**59**, o280–o282.